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Nickel Recovery from Electroplating Rinsewaters by Electrodialysis

Risdon Mfg. Co.
Waterbury, CT

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16. ABSTRACT
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A cost estimate based on the data obtained during the demonstration indicates that 95% of the nickel lost from untreated rinses could be recovered and that ancillary benefits in sludge disposal, use of treatment chemicals, etc. could be realized. Payback periods of less than 18 months are anticipated for commercial units.

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FOREWORD

When energy and material resources are extracted, processed, converted, and used, the related pollutional impacts on our environment and even on our health often require that new and increasingly more efficient pollution control methods be used. The Industrial Environmental Research Laboratory-Cincinnati (IERL-Ci) assists in developing and demonstrating new and improved methodologies that will meet these needs both efficiently and economically.

This report is a product of the above efforts. It was undertaken to demonstrate the effectiveness and economic feasibility of using electrodialysis for nickel salt recovery and pollution control on a Watts-type nickel plating line under actual plant conditions. The nickel salts recovered from the rinse solutions were concentrated 20-fold by the electrodialysis treatment and could be returned directly to the plate tanks for reuse. The results of the report are of value to R & D programs concerned with the treatment of wastewaters from various metal finishing, non-ferrous metal, steel, inorganic, and other industries. Further information concerning the subject can be obtained by contacting the Nonferrous Metals and Minerals Branch of the Energy Pollution Control Division.

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ABSTRACT

A program to demonstrate the feasibility of metal salt recovery and pollution control on a Watts-type nickel plating line by electrodialysis was conducted at Risdon Manufacturing Co., Waterbury, CT. Each of two reclaim rinse tanks, arranged in series following plate tanks, was treated by recirculating the rinse solutions through separate electrodialysis stacks. The first rinse solution was maintained at 2-5 g/l nickel and the second rinse held at 0.3-0.4 g/l nickel over several months of plating operations. The nickel salts recovered from the rinse solutions were concentrated 20-fold by the electrodialysis treatment and could be returned directly to the plate tanks for reuse. Several operational problems are discussed and recommendations made. It is concluded that electrodialysis can be a useful and economically viable process for the treatment of at least some types of electroplating rinses. Both plating-metal recovery and pollution control are accomplished.

A cost estimate based on the data obtained during the demonstration indicates that 95% of the nickel lost from untreated rinses could be recovered and that ancillary benefits in sludge disposal, use of treatment chemicals, etc. could be realized. Payback periods of less than 18 months are anticipated for commercial units.

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Thanks are also owed to Fred Ellerbusch and Mary K. Stinson of the Environmental Protection Agency for their generous encouragement and support.

SECTION 1

INTRODUCTION

Electrodialysis (ED) is a membrane process that can be used for the separation, removal, or concentration of ionized species in water solutions. These operations are accomplished by the selective transport of ions through ion-exchange membranes under the influence of an electrical potential applied across the membrane. Ion-exchange membranes, permeable to either anions or cations, but not both, are thin sheets of ion-exchange material normally reinforced by forming on a synthetic fabric backing. They range between 0.1 to 0.6 mm in thickness and are available in standard sheets up to 1 x 1.5 meters. As in the case of particulate ion-exchangers, the resin matrix is usually copolymerized styrene-divinylbenzene, and exchange capacity is imparted by sulfonic acid groups (cation-selective membranes) or quaternary ammonium or pyridinium groups (anion-selective membranes). The terms cation-exchange, cation-selective, and cation-permeable are used interchangeably in membrane terminology as are the corresponding terms anion-exchange, anion-selective and anion-permeable.

In the usual configuration employed for electrodialysis, many, on the order of hundreds, alternating anion-selective and cation-selective membranes are arrayed in parallel between two electrodes to form an ED multicell or "stack". Especially designed spacer/gaskets separate the membranes by forming leak-tight, flow-directing compartments or cells between adjacent membranes. The solutions to be treated are distributed to and collected from these cells by two internally manifolded hydraulic circuits, one for the ion-depleting or diluting cells and one for the alternating ion-receiving or concentrating cells. The repeating stack unit of a cation-selective membrane, a diluting spacer, an anion-selective membrane, and a concentrating spacer is termed a cell-pair, and the size and demineralizing capacity of electrodialysis equipment can be characterized by indicating the number of cell-pairs comprising a multicell.

The passage of a direct current through the electrodialysis stack causes the anions and cations in the raw process solution fed to the diluting cells (the feed) to move in the direction of the anode and cathode, respectively. Because of the alternating

membrane arrangement they leave the diluting cells and accumulate in the concentrating cells. This process is shown schematically in Figure 1 where the concentrating cells are odd numbered and the diluting cells have even numbers. As indicated, positive cations are attracted to the negative cathode and pass from the diluting compartments, through the cation-selective membranes forming the cathode side of the cell, into the concentrating compartments where they accumulate, since their further transport is prevented by anion-selective membranes on the cathode side of the concentrating cells. Anions move in the opposite direction, passing through the anion-selective membranes and being excluded by the cation-selective membranes. The partially deionized effluent from the diluting cells may be suitable for use after a single pass through the stack or recirculated for further demineralization. The solution introduced to the concentrating cells is usually the same process stream as that fed to the diluting cells, but only 20-25% of its volume, and is eventually discarded at 3 or 4 times the original concentration having acquired the ions removed from the solution passing through the diluting cells. A separate solution, which may also be drawn from the raw stack feed, is used to rinse the electrode compartments and remove the gases formed by the electrode reactions. It may be recirculated or pumped on a once-through basis and is usually acidified to prevent scaling. Use of the multicell concept makes it possible to obtain a deionizing effect equal to many times the electrical equivalents passed between the electrodes.

In the late 1940's, the development of highly selective and physically strong synthetic ion-exchange membranes made feasible commercial application of the electrodialysis multicell (1, 2). The first and still largest use of ED is for desalting brackish water; the second major application is deashing whey. Besides these now conventional uses, in which the desired product is the deionized feed to the diluting cells, an interesting and important application of ED is the deliberate use of the concentrating steam to concentrate and recover ionic constituents from the waste or process water fed to the stack. In this modification the recovered concentrate can also be considered a product solution from the electrodialysis treatment, and the recovered material is available for reuse in the original process or for further treatment and/or disposal. In the latter case subsequent handling of the recovered solution is substantially simplified, since it is typically 1-2% of the original volume. Electrodialytic concentration of sea-water is widely used in Japan in conjunction with solar evaporation to produce table salt and has great potential utility in the electroplating and metal finishing industries both for pollution control and metal recovery. The Japanese have extended their work on concentration by ED to exploratory studies on waste liquors from copper and nickel plating operations (3), and laboratory tests in this country (4) have also indicated the possibility of electrodialyz-

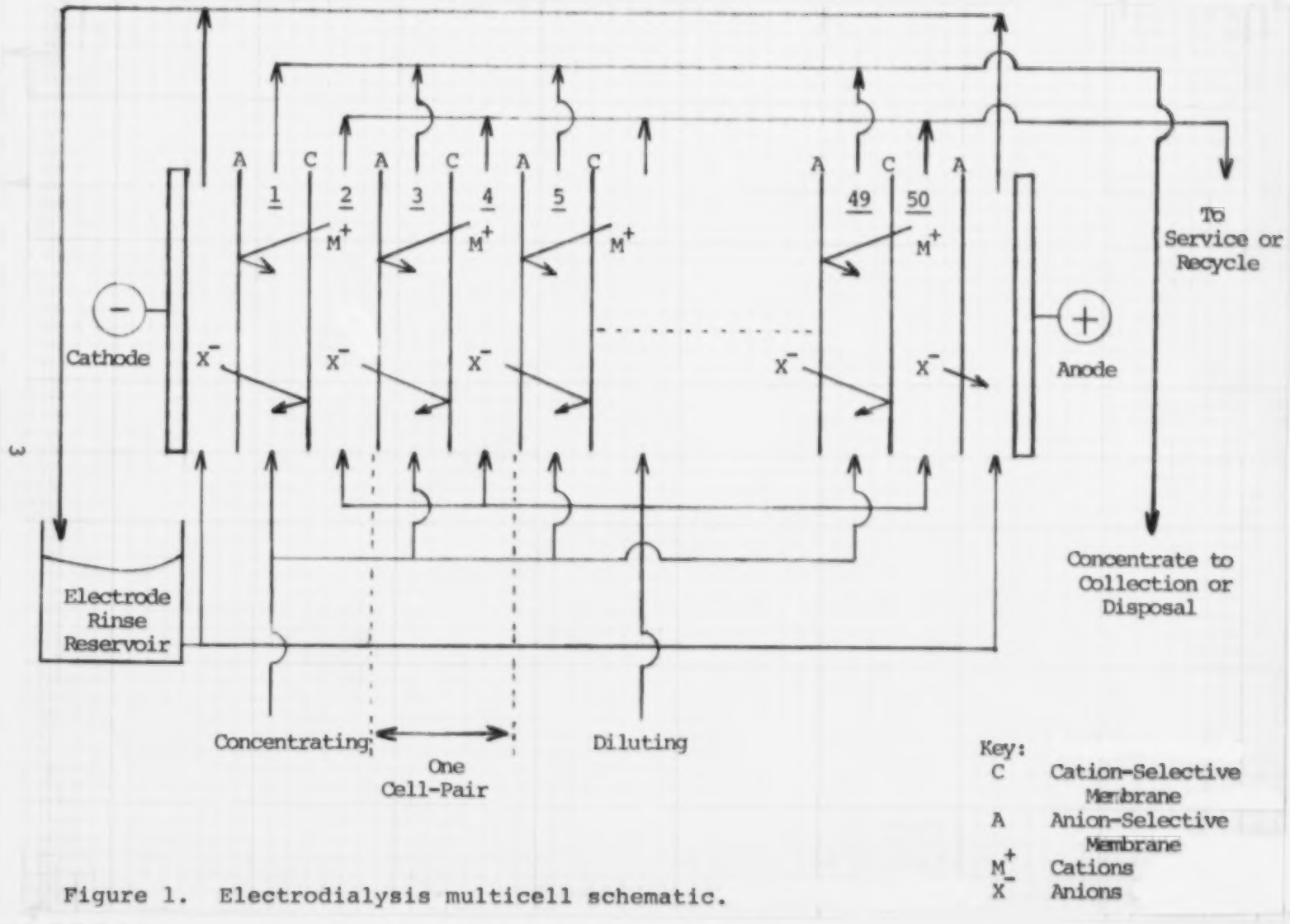

Figure 1. Electrodialysis multicell schematic.

ing nickel solutions. Another recent laboratory investigation has looked at the electrodialysis of a copper cyanide plating bath rinse for recovery of plating chemicals and elimination of toxic discharges (5). Our own preliminary studies have indicated that nickel can be recovered as a valuable product solution from a simulated plating rinse bath at many times its concentration in the bath by using especially designed electrodialysis multi-cells and appropriate operating conditions (6). Based on these laboratory data, a prototype ED unit was constructed for a field test and demonstration program at Risdon Manufacturing Co., Waterbury, Connecticut.

SECTION 2

CONCLUSIONS

The field test program demonstrating the recovery of nickel from plating bath rinse waters by electrodialysis has shown that ED can be a useful and economically viable process for the treatment of at least some types of electroplating rinses, specifically those from Watts-type nickel plating lines. The process is capable of reducing nickel content in plating line effluents to the ppm range, can recover 95% of the nickel salts lost with conventional rinsing techniques and substantially reduce other raw material usage, can minimize the effort and space required for operation of standard destruct equipment, and can return a concentrated nickel solution directly to the plate tanks.

Electrodialysis treatment of a reclaim rinse immediately following the nickel plate tanks held the rinse concentration at 2-3 g/l nickel. A second reclaim tank in series with the first was maintained at approximately 0.3 g/l nickel. Lower concentrations appear to be easily attainable with appropriate equipment sizing. Metal losses via drag-out to the destruct system following the reclaim rinses were drastically reduced compared to those expected with a countercurrent or unmodified reclaim rinsing sequence. For plating operations similar to the demonstration line yearly savings are estimated to be at least $16,000 in raw materials with a concomitant decrease of about 2,300 kg (5,000 lbs.) in sludge from pollution control facilities. Net cost savings should increase as chemicals become more expensive and pollution regulations more stringent. Depending on the operation of the plating line, additional savings may be realized on water usage, storing and handling of chemicals, improved rinsing, and additive recovery.

In contrast to reverse osmosis, ED can concentrate the nickel 50- to 100-fold, permitting direct return to the plating solutions without the necessity of an extra evaporation step or increase in plate tank evaporation rate. The membranes employed are mechanically stronger and more resistant chemically than typical reverse osmosis membranes. The concentration of the reclaim rinse can be held at any desired value and the recovery rate adjusted to remove nickel to match any drag-out rate. Electrodialysis is a continuous process and requires no inter-

ruption for regeneration of ion-exchange material or disposal of regenerating solution as do conventional ion-exchange systems.

The program has also demonstrated that maintenance and operation of electrodialysis equipment by plating room personnel are completely satisfactory with a minimum of training. Other than for start-up and shut-down procedures only intermittent attention by the operator is required and much of his time can be spent on other duties.

SECTION 3

RECOMMENDATIONS

The success of the electrodialytic treatment of nickel rinse solutions described in this report suggests that further investigations aimed at broadening its applicability would be desirable. Recommendations are made that demonstration programs be carried out on other types of nickel plating systems, particularly on those systems that use organic brighteners or other classes of additives not employed on the test line at Risdon. Extension of the technique to additional plating solutions, i.e. copper and chromium, would be of considerable interest. Simple salt or "acid" baths are the obvious choices for initial efforts. Demonstration of ED on plating lines using countercurrent rinsing and/or automatic barrel handling would also be valuable in estimating the potential savings in water and space requirements, in defining system versatility, and in generalizing experience with the process. Improved maintenance schedules and more accurate estimates of costs and component life would be developed.

At least two employees familiar with the individual plating operation should be trained in electrodialysis technology to insure full-time monitoring capability. Responsibility for the ED equipment must be recognized as an integral part of their job and an appropriate percentage of their time assigned to recovery operations. Adequate filtration of the influent to the ED stack is a required pretreatment, and some cooling may be necessary. Wide fluctuations in ambient temperature should be avoided. Besides the safety precaution in the interconnection of the stack current/feed pump circuitry, additional DC cutoffs based on flow attenuation and feed conductivity are desirable.

SECTION 4

EQUIPMENT AND OPERATION

The electrodialysis prototype module constructed by Chemical Recovery Systems for the demonstration program was based on a 50 cell-pair electrodialysis stack. A photograph of the module is shown in Figure 2. The overall stack dimensions were 63.5 cm x 35.6 cm x 45.7 cm (25" x 14" x 18") including sealing clamps, tie-rods, and piping connections. Actual size of the membranes and spacers was 55.9 cm x 30.5 cm (22" x 12") with approximately 65% of the membrane area used for ion transfer. Neosepta membranes supplied by the Tokuyama Soda Co. were used. The spacers delineated an S-shaped flow path which combined the advantages of the more common sheet flow and tortuous path designs. Diluing and concentrating cells were 2.2 mm and 1.2 mm thick, respectively, and otherwise differed only in the size of the inlet and exit slots connecting the individual cells to the manifolds. The cells contained an open plastic netting in the flow path for membrane support and turbulance promotion. Each end of the stack terminated at similar electrode cells formed by an anion-selective membrane and a stainless steel cathode or a platinized titanium anode. When operating, a hydraulically isolated electrode rinse solution of acidified sodium sulfate was recirculated through the two electrode cells from a small reservoir.

The stack was mounted on a 106.7 cm x 116.8 x 68.6 cm (42" x 46" x 27") mobile frame that also carried all equipment and controls necessary for operation of the prototype. The entire unit could be wheeled up to the rinse tank to be treated and be ready to operate after connection to a 3-phase, 230 volt electrical source and inlet and outlet hoses for the stack feed solution (the rinse solution from which nickel was to be recovered). A sketch of the several hydraulic circuits in the module is shown in Figure 3. Flow was from the rinse tank, through a cartridge filter, strainer, and flowmeter, to the diluting or feed compartments of the ED multicell, and then returned to the rinse tank. Panel-mounted meters indicated feed stream temperature, pressure both at filter and stack inlet, and conductivity of the rinse solution. Current density through the stack was controlled by adjusting a voltage control on the power supply and monitored via DC volt and ammeters. Full-wave, 3-phase rectification was used to provide a direct current with less than

Figure 2. Prototype electrodialysis unit.

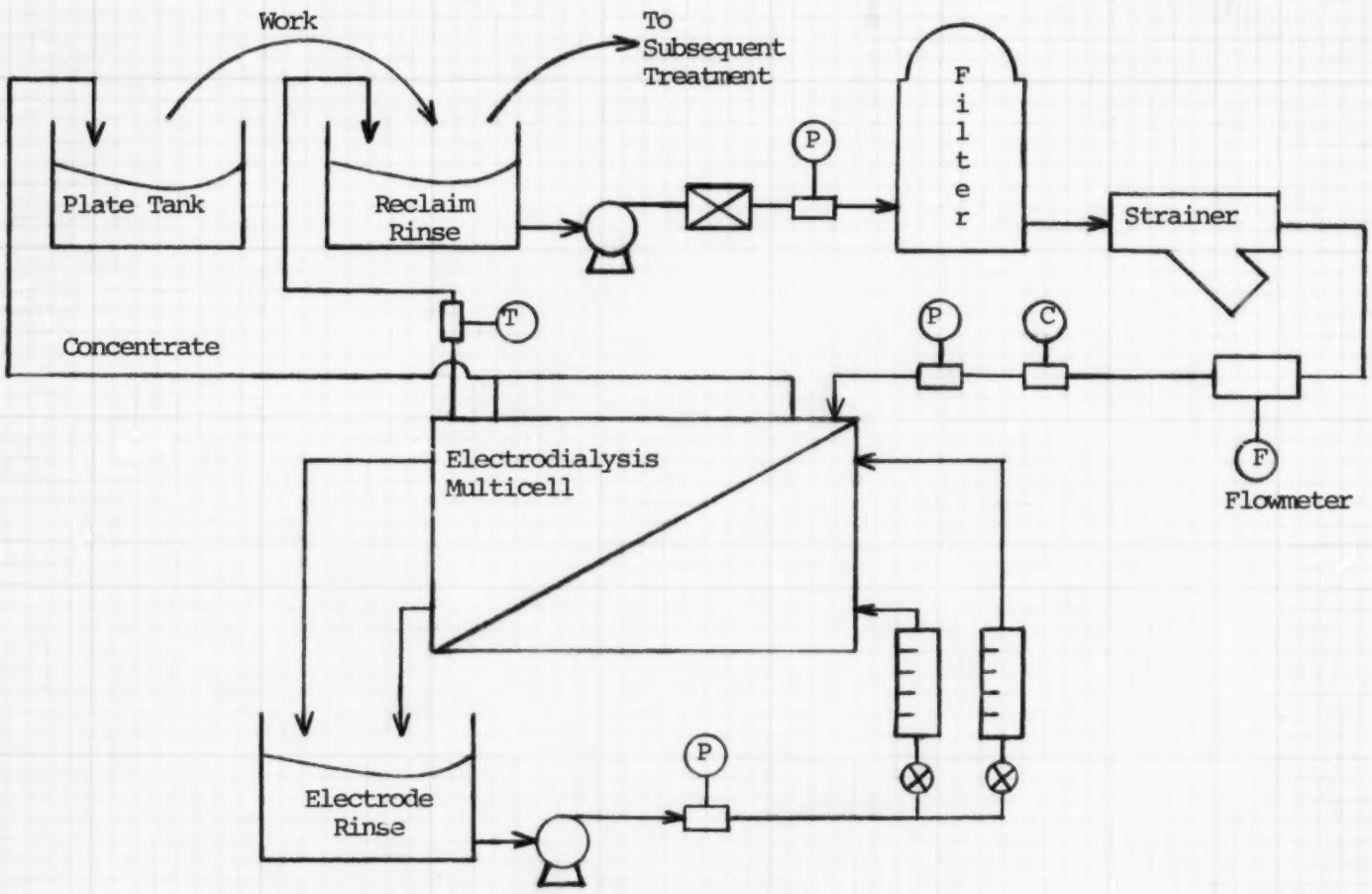

Figure 3. Hydraulics of electrodialysis module.

5% ripple. Electrical outlets and switches for feed and electrolyte pumps and line-powered instruments were incorporated into the power supply package. Circuits were arranged so that stack DC could not be activated unless the feed pump was running, and a single main switch incorporating a circuit breaker controlled all power to the unit. There was no pumped concentrate through the concentrating cells. The concentrate solution was produced only from the water of hydration associated with the movement of recovered ions into the concentrate or product solution and the electroosmotic water transport. This arrangement simplified the operation of the ED module and produced high product concentrations.

The 50 cell-pair prototype unit was installed at Risdon on a Watts-type nickel barrel-plating line and operated to recirculate through the electrodialysis stack a still or reclaim rinse solution (Rinse I) that immediately followed the plate tanks. A second ED unit was subsequently installed on a second reclaim rinse (Rinse II) following the first rinse in order to assess the possibilities of achieving a closed loop plating operation by use of electrodialysis. The second ED unit (Unit II) was similar to the first ED unit (Unit I) and operated in the same manner in recirculating Rinse II. However, since the rate of nickel drag-in to the second rinse was much less than that to the first rinse, Unit II multicell contained only 20 cell-pairs, operated at lower current densities, and recovered substantially less nickel than Unit I. A diagram of the plating line and the location of the electrodialysis units is shown in Figure 4.

Drag-in to Rinse I from the plate tanks was 300-500 grams per hour nickel at a plating rate of 2-3 barrels per hour, while nickel drag-in to Rinse II was only 10-20 grams per hour at the lowest concentrations maintained in Rinse I. Concentrated product from both units was collected in a small external reservoir and the combined concentrates periodically returned to the plate tanks. Product volume varied between 30-40 liters per day for Unit I and 4-6 liters per day for Unit II, depending chiefly on the required current density.

Figures 5 and 6 are photographs of the two electrodialysis units as they appeared during the course of the field test program. In operation, the units were turned on in the morning about an hour before the first barrel went through the line and run during the one or two shifts scheduled for the day. Before start-up the solutions in the plating tanks were replenished, if necessary, from Rinse I, and any evaporation and drag-out losses both from Rinse I and II were made up with city water. During the course of the day the two rinse concentrations were monitored with conductivity meters and the current density through the electrodialysis multicells manually adjusted to vary the rate of nickel removal and maintain the desired nickel concentration level in the rinses. Samples of the rinse

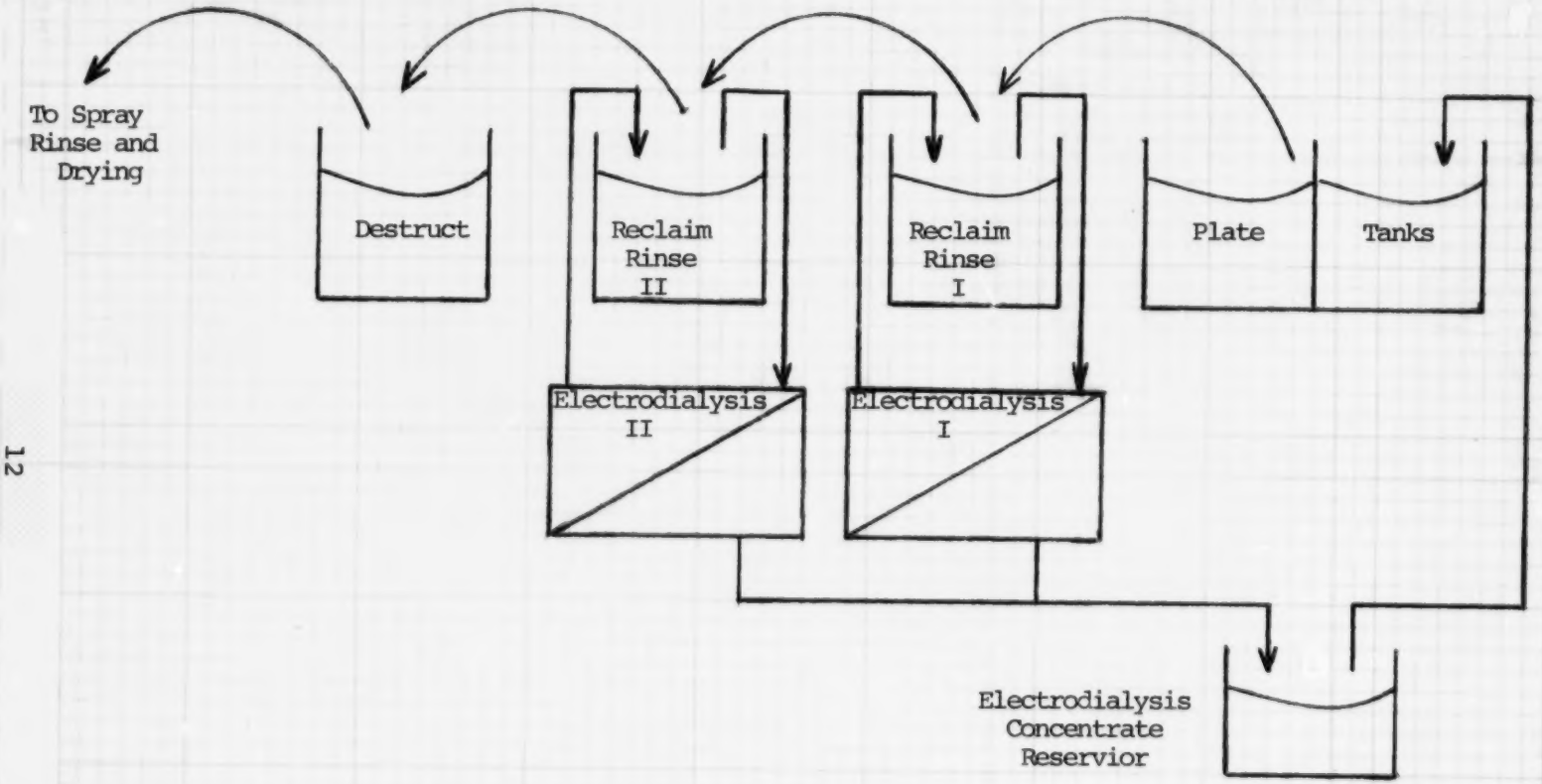

Figure 4. Electrodialysis treatment of nickel plating line.

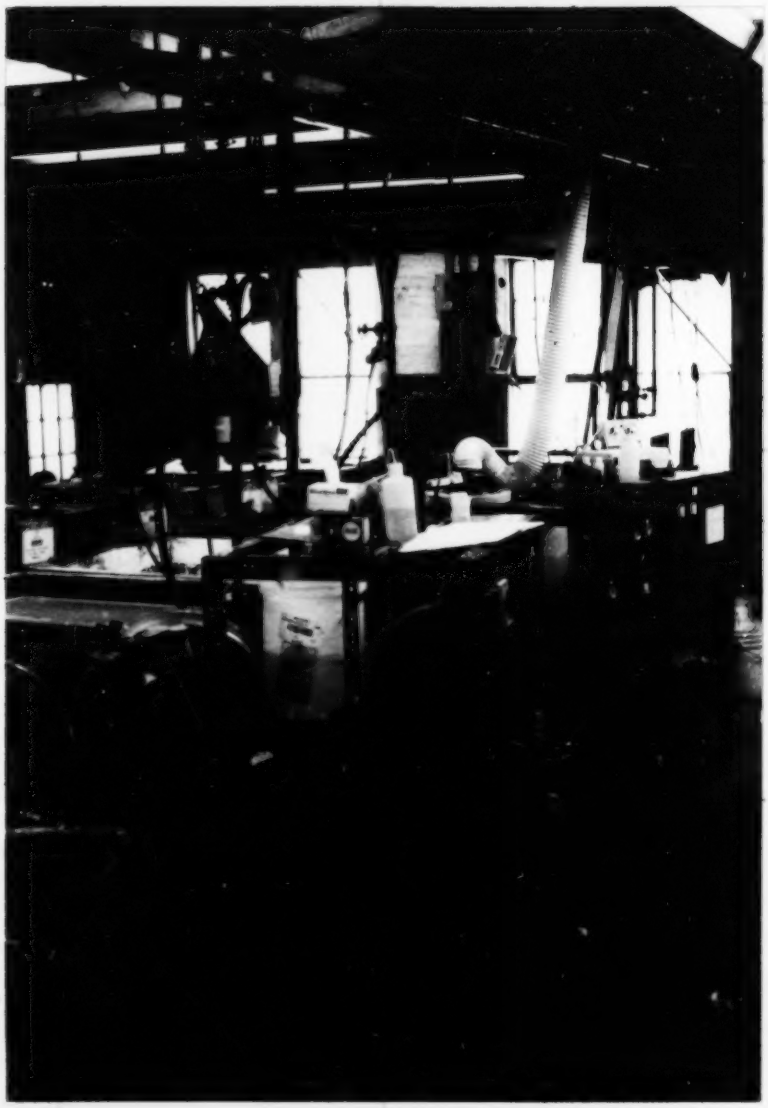

Figure 5. Field test site.

Figure 6. Field test electrodialysis units.

and product solutions for each unit were taken for analysis each operating day just before shutdown. Routine running of the Units was left to the plating foreman as soon as he became familiar with their operation. They did not require continuous attention and total time necessary was between 30-40 minutes a day, 10-15 minutes for start-up and about 5 minutes for shutdown and each of several relatively brief checks and adjustments during the day. Filter cartridges were changed when required as indicated by feed flow attenuation and/or pressure increase. If necessary or desirable, operation was also interrupted for modifications or adjustments on the modules or when the membrane stacks were exchanged for cleaning, to introduce design changes, or to correct malfunctions. However, these maintenance operations could usually be performed before or after daily plating began. In either event, they normally took no longer than 15-30 minutes.

SECTION 5

RESULTS AND DISCUSSION

First Series of Tests

Unit I, as initially installed at Risdon incorporated only a 40 cell-pair membrane stack in order to insure that excess stack current and pumping capacity were available during the early stages of the field test. Closed-loop operation was not started until the second test series, with the installation of Unit II. Thus, instead of the work flow indicated in Figure 4, the temporary sequence was plate tanks to Rinse I undergoing ED treatment, then to destruct, final rinse, and spray rinse. This interval was regarded as a break-in and debugging period with emphasis on operator familiarization and training. Drag-out volume and typical plate concentrations were determined, and tentative operating procedures for the Unit were written to conform to existing plating line practices. The ED equipment initially was operated only during the first of two shifts, holding the rinse concentration steady during first shift operation and then allowing it to rise during the second shift. Shortly after start-up a second operator was sufficiently trained so that he could monitor the Unit during any scheduled second shift and handle shut-down procedures.

Table 1 is a summary of the pertinent data obtained during this series of tests, which was performed over a three-month period. The feed and product concentrations were determined from samples taken at the end of the daily operations, and the pH was also measured from these samples. An operating day was any day (starting with the 7:00 A.M. shift) during which the electrodialysis module was functioning to treat the rinse solution. If the unit was inoperative for any reason, an operating day was not recorded. After two ED units were installed, an operating day was logged if either unit was functioning. The reported temperature is the range recorded for the rinse solution during the day with the increase resulting from the drag-in of hot plating solution and the heat generated by the recirculation pumps and stack current. The plating room itself was poorly heated, and, especially over weekends, the ambient temperature could drop quite low. This caused some problems with dimensional changes in stack components, solution resistance,

TABLE 1. ELECTRODIALYSIS OF NICKEL RECLAIM RINSE I
FIRST TEST SERIES

Operating Day	Amps/Volts	Temp., °F	Feed, g/l Ni	Product, g/l Ni	pH Feed/Prod.	Time run, Hours
1	3/17	69-83	4.5	65	6.5/-	5
2	7/40	70-90	4.2	89	-	8
3	9/50	77-87	5.2	87	6.8/-	8
4	10/50	60-90	13	86	-	9
5	10/40	69-88	16	79	-/5.5	7
6	12/103	58-87	5.1	87	7.0/-	8
7	10/75	73-95	5.9	92	7.0/5.8	7
8	10/58	68-94	9.1	90	6.7/5.6	6
9	10/60	70-93	9.7	89	6.7/5.6	8
10	10/90	67-83	5.5	94	6.9/5.6	5
11	10/100	66-90	6.5	88	6.7/5.5	7
12	6/26	72-79	33	68	6.0/5.2	3
13	10/92	68-80	9.5	76	6.0/4.5	8
14	10/90	66-93	7.6	75	6.3/5.0	9
15	8/90	74-83	-	-	-	7
16	15/34	71-72	19	76	6.5/5.3	2
17	10/30	64-74	19	72	6.4/5.3	6
18	10/35	70	18	60	6.4/5.4	9
19	8/38	59-74	5.7	56	6.9/5.3	9
20	8/40	70-75	8.3	64	6.8/4.7	15
21	10/40	64-80	13	67	6.6/4.9	15
22	10/35	62-	21	78	6.3/5.1	5
23	10/52	58-72	9.3	64	6.6/5.4	15
24	7/66	68-72	8.9	57	6.7/5.0	14
25	9/95	64-73	10	60	6.5/4.9	15
26	8/115	66-72	14	57	6.4/1.8	15
27	10/76	64-74	10	70	6.3/2.4	14
28	10/70	68-73	9.6	66	6.3/4.5	9
29	10/105	56-77	5.9	69	6.9/3.0	13
30	10/90	64-76	7.9	45	6.7/4.8	15
31	10/140	64-72	-	-	-	10
32	8/77	67-70	6.0	103	6.9/5.4	9
33	8/60	58-72	6.3	38	6.9/6.0	12

(continued)

TABLE 1. (continued)

Operating Day	Amps/Volts	Temp., °F	Feed, g/l Ni	Product, g/l Ni	pH Feed/Prod.	Time run, Hours
39	8/40	72-85	20	83	5.6/4.7	8
40	10/51	50-84	17	90	6.2/5.4	14
41	8/36	70-85	18	71	6.2/5.5	10
42	10/50	54-85	12	88	6.0/5.2	11
43	10/35	64-85	10	83	6.5/5.6	14
44	12/82	56-88	9	30	6.9/1.0	9
45	11/71	57-80	3.4	83	6.9/5.4	6

and crystal formation. The latter will be discussed later. Values reported for amperage and voltage were those reported by the operator as required to maintain a constant nickel concentration in the rinse, as indicated by solution conductivity, under the existing stack and rinse tank conditions. Total operating time was 418 hours and the average feed concentration over the period was 11.4 g/l nickel. This concentration level was achieved by part-time electrodialysis treatment only and was substantially improved as the ED equipment became more fully operational later in the demonstration program. Nevertheless, it was a marked improvement over the concentration of the untreated rinse that, for example, increased to 33 g/l nickel from 6.5 g/l during a four day interruption of the electrodialysis treatment. A separate test gave an increase from zero (city water) to 21 g/l nickel, also in four days. Calculations indicated that the equilibrium concentration in the rinse would reach about 50 g/l nickel if used as a simple reclaim tank to replenish the plate tanks. The average product concentration was 73 g/l nickel, and at a current of 9.5 amperes, product accumulated at a rate of approximately 3.5 liters per hour , giving a coulomb efficiency of about 60%. On an eight-hour day basis, this returned 28 liters (7.4 gallons) containing 2.04 kg of nickel metal or about 20 pounds of nickel salt (as $NiSO_4.6H_20$) to the plate tanks. This volume of solution was easily accommodated by the plate tanks which still required periodic replenishment from the rinse solution to make up evaporative losses. It took 2-3 hours for the stack product to reach a steady-state concentration under a given set of operating conditions, and any interruption or change in the operations affected the final product concentration. Since adjustments in current, brief shutdowns for filter changes, replenishment of the plate tanks with subsequent dilution of the rinse, and other experimental operating changes were normal occurrences, the observed variations in product concentration were to be expected.

After several days operation of the ED module, it became apparent that the temperature of the rinse tank could approach 100°F towards the end of an eight-hour shift. Thus, for 12-15 hours of continuous plating the manufacturers recommended maximum temperature of 40°C (104°F) for the membranes would probably be exceeded and the ED unit have to be shut down prematurely. In practice, an operating temperature maximum of 35°C (95°F) was specified to provide a safety margin. Consequently, a flat heat exchanger fabricated from loops of 3/4" polyethylene tubing was installed on the bottom of the rinse tank. Water used for the final spray rinse hopper was diverted to flow first through the tubing coils, and its volume was sufficient to keep the rinse temperature at acceptable levels for long periods of time. This cooling arrangement did not require any additional water usage and was automatically activated if the plating line was operating.

A more serious problem was the formation of large deposits of greenish-blue, hot-water soluble crystals in the product cells and recovery tubing after a few weeks of module operation. These crystals could completely block the flow of product solution and accumulate in the product cells to the point where their expansion would attenuate the flow in the feed cells. If crystals were present, a strong smell of ammonia was detected when the stack was disassembled for inspection and cleaning. Similar results were obtained in the laboratory using a duplicate ED stack and samples of Risdon plate solution. From these observations and available data on plate tank additives, the crystals were assumed to be nickel ammonium sulfate, and this identification was confirmed by elemental analysis on material collected from blocked cells. Origin of the crystals was from the ammonium hydroxide that had been added to the plating solutions to control pH. This led to formation of the nickel-ammonium double salt which subsequently was concentrated by the electrodialysis treatment to the point of insolubility at plating room temperatures. It is only one-tenth as soluble as nickel sulfate itself. Salts of comparable or lower solubility would also limit the ability of ED to concentrate them. Indeed, one of the major concerns in electrodialytic desalting of water is the formation of scale in the concentrating cells due to the accumulation of species with limited solubility. Disassembly of the stack and inspection of the membranes following incidences of crystallization showed severe erosion on the product cell side of the anion-exchange membranes, producing a roughened surface which sometimes exposed the cloth backing. Many membranes developed leaks, but those less severely attacked were returned to service after testing. Crystallization within the membrane had probably cracked off the surface resin. Fortunately, use of ammonia in the Risdon plate tanks was in the process of being replaced by other pH control methods, so the problem gradually disappeared as the ammonium salts were lost from the plating line. Subsequent isolated additions of ammonia did not affect the operation of the ED Unit, and a series of nitrogen analyses indicated that the plate tanks normally contained 1.5-3 g/l of total Kjeldahl nitrogen without any apparent detrimental effects. If necessary, the difficulty could have been at least partially mitigated by adjustments in the allowable current density and stack size to limit product concentration without decreasing total nickel recovery.

Also associated with the presence of ammonium salts in the plating solutions was the appearance of a pungent, irritating odor in the electrolyte reservoir, accompanied by heavy white fumes. Although no analyses were performed, the odor was ascribed to nitrogen trichloride formed by reaction at the electrodes since an industrial preparation of NCl_3 is via the electrolysis of ammonium chloride in an acid solution. This hypothesis is consistent with the fact that the odor and fumes disappeared as the occurrence of crystals decreased and the use

of ammonia was curtailed. A small elephant-trunk vent duct suspended over the electrolyte tank effectively removed the odor when present, along with the other gases formed at the electrodes.

Second Series of Tests

The second period of the field test began when Unit II was added to the plating line on Rinse II and the rinsing sequence depicted in Figure 4 followed. The second series of tests covered a six-month period and it was planned, as far as possible, to operate both ED modules whenever the plating line was running to minimize the quantity of final nickel drag-out to the chemical destruct system and to approach as nearly as possible a closed-loop plating line. Data for Unit I for the second test series are given in Table 2. Over the entire period Rinse I feed solution was held at an average of 5.7 g/l nickel with the higher individual values occurring in the middle of the test period when difficulties were encountered with the conductivity monitor. The product concentration continued to be sensitive to any change in operating conditions and vary with fluctuations in feed flow rate, pH, temperature, current density, or rapid dilution of the reclaim tank. However, it averaged 61 g/l nickel for an 11-fold concentration ratio, and when the module ran undisturbed on rinse concentrations in the 2-3 g/l range, the product concentration was more than 20 times that of the rinse solution. The volume of product was slightly higher than in the previous period, returning about 5 liters (1.3 gallons) per hour to the plate tanks. This increase was due to the installation of a 50 cell-pair stack in order to provide increased removal capacity at lower stack current and make it easier to cope with variations in the rate of plating, drag-out volume fluctuations, temperature of the rinse solutions, and rinse tank usage. Since the plater was following his normal salt addition schedule for the plating solutions during most of this period, the nickel concentration in the plate tanks was substantially increased by the returned ED product. At one point it had built up to 141 g/l. This was 1.6 times the normal plate strength and significantly increased nickel drag-out from the plate tanks. Overall, during this period Unit I was on-line 80% of the time the plating line was operating with several of the non-operating periods occurring simply because the operator was unavailable. For the last two months the on-line percentage rose to 95%. Operating time was 949 hours for the period with a total of 1,367 hours for Unit I from the start of the test.

Considerable effort during this period was aimed at improving the operation of the prefilters immediately ahead of the membrane stack on Unit I. For most of the time there was no continuous independent filtration of the plate solutions and significant amounts of iron and nickel hydroxides built up in

TABLE 2. ELECTRODIALYSIS OF NICKEL RECLAIM RINSE I
SECOND TEST SERIES

Operating Day	Amps/Volts	Temp., °F	Feed, g/l Ni	Product, g/l Ni	pH Feed/Prod.	Time run, Hours
1	14/96	64-92	3.4	72	6.9/2.5	9
2	7/8	70-72	-	-	-	4
3	12/135	64-89	-	-	-	7
4	8/107	74-84	4.1	32	6.8/2.3	6
5	10/48	72-89	5.6	80	6.7/5.7	8
6	12/72	69-90	3.5	83	6.0/3.6	9
7	10/90	65-78	3.4	92	7.0/5.6	9
8	5/105	46-75	3.6	84	7.1/5.7	6
9	9/70	68-82	3.9	86	6.9/5.6	8
10	9/80	66-93	3.8	68	7.1/1.8	9
11	10/90	72-88	-	-	-	7
12	9/125	58-82	3.7	66	3.5/1.9	4
13	8/125	74-85	2.4	66	6.2/1.7	8
14	9/84	89	3.7	83	6.6/4.9	9
15	6/95	60-90	3.7	77	6.7/5.2	9
16	7/95	69-93	3.4	44	6.7/5.9	8
17	10/62	72-82	3.1	62	6.2/4.7	4
18	7/90	68-80	2.0	47	4.2/6.1	3
19	10/55	68-88	3.9	65	7.1/5.9	8
20	9/63	70-90	3.8	56	7.2/4.8	7
21	8/95	68-86	3.0	23	7.3/1.6	8
22	10/95	66-84	4.7	63	7.1/2.3	8
23	7/90	53-85	4.8	57	7.0/5.4	8
24	12/60	69-91	4.3	71	6.0/4.7	9
25	9/87	70-85	4.2	56	6.7/2.6	9
26	9/100	78-94	4.4	49	7.0/2.7	9
27	9/80	82-94	5.0	63	6.9/4.2	7
28	8/90	68-90	6.1	60	6.8/2.2	15
29	7/100	70-82	4.0	60	7.1/1.7	8
30	10/95	66-91	4.1	79	7.0/5.7	6
32	9/90	88	3.3	74	6.5/3.3	8
33	8/42	68-80	4.4	60	6.3/6.1	9
34	9/60	56-90	2.0	61	6.9/6.1	13

(continued)

TABLE 2. (continued)

Operating Day	Amps/Volts	Temp., °F	Feed, g/l Ni	Product, g/l Ni	pH Feed/Prod.	Time run, Hours
36	12/30	72-90	5.5	69	6.8/5.8	13
37	10/35	76-98	5.5	67	7.0/5.6	8
38	12/30	66-86	13	64	6.8/5.3	12
39	10/60	65-90	3.9	65	7.0/1.6	15
40	9/40	70-96	3.4	39	6.9/6.1	15
41	8/45	70-88	3.4	43	7.0/6.1	7
42	9/43	62-84	4.4	59	7.1/5.8	10
43	9/55	69-88	4.9	69	7.1/5.1	8
44	8/52	72-88	2.9	59	7.4/5.7	9
45	10/65	66-94	3.1	72	7.2/4.9	8
46	5/75	73-77	2.9	44	7.2/1.8	9
47	9/65	65-96	5.5	53	6.9/5.2	9
48	9/80	70-95	3.5	62	7.2/5.1	10
49	5/65	68-74	4.9	64	6.7/3.5	5
50	8/60	69-92	-	47	-/2.7	14
51	8/70	70-93	13	53	6.8/5.6	9
52	10/40	68-90	12	66	6.9/5.6	12
53	9/40	75-90	8.9	63	6.8/5.7	14
54	9/30	72-92	8.2	54	7.0/5.7	14
55	9/30	68-86	8.1	68	7.0/5.3	6
56	8/25	72-88	9.5	55	6.9/5.8	6
57	9/71	69-89	8.4	52	7.0/1.6	15
58	8/40	8	-	-	-	16
59	9/32	67-83	8.2	75	6.8/5.7	10
60	10/40	71-83	8.0	76	6.6/5.6	16
61	10/40	70-88	8.4	65	7.0/5.7	8
62	8/40	76-92	11	53	6.9/5.7	9
64	9/45	80-89	11	59	7.0/5.6	6
65	5/85	88	7.1	46	7.0/1.5	9
66	8/45	68-80	15	43	6.7/5.8	7
67	9/75	67-87	10	42	6.9/2.4	8
68	8/43	68-74	6.6	57	7.0/5.4	10
69	8/43	69-74	8.5	61	6.9/5.4	8
71	12/46	63-89	7.0	81	6.7/5.5	6

(continued)

TABLE 2. (continued)

Operating Day	Amps/Volts	Temp., °F	Feed, g/l Ni	Product, g/l Ni	pH Feed/Prod.	Time run, Hours
72	9/32	77-90	9.2	70	6.6/5.3	14
73	7/65	76-85	-	-	-	6
74	9/35	74-90	4.4	55	6.7/5.5	17
75	9/27	72-90	9.6	70	6.2/5.2	9
76	8/20	77-93	2.7	61	6.8/5.3	9
77	9/36	69-90	5.2	74	6.4/4.0	8
78	8/70	73-88	6.2	48	6.2/2.4	7
79	9/40	68-84	7.1	63	6.4/4.8	9
80	9/45	72-83	5.1	80	6.1/2.5	9
81	9/41	74-80	7.1	78	6.5/5.2	9
82	7/47	70-87	2.6	61	4.6/2.3	10
83	9/48	74-96	3.9	58	6.6/2.1	9
84	9/87	65-87	7.2	53	6.7/1.4	6
85	9/76	67-90	6.4	61	6.3/1.6	7
86	15/106	74-90	-	-	-	7
87	11/29	70-92	2.6	54	6.6/5.9	11
88	10/32	74-98	3.5	52	6.9/5.6	9
89	10/25	72-86	2.1	20	7.4/2.3	9
90	9/31	73-95	3.0	44	7.0/6.0	9
91	10/25	76-87	5.4	62	6.9/5.8	13
92	9/34	74-98	7.1	43	6.4/2.6	15
93	9/35	65-87	3.8	65	5.7/2.7	9
94	11/35	82-91	4.5	70	6.4/5.4	15
95	12/30	77-100	5.9	51	6.6/5.4	8
96	12/37	80-102	3.2	66	6.5/5.3	6
97	10/25	84-102	5.7	48	6.5/3.2	7
98	9/30	70-80	5.4	57	6.3/2.5	15
99	12/30	81-90	6.0	58	6.1/3.5	15
100	9/20	80-98	7.0	55	5.3/3.6	15
101	12/20	80-95	7.1	66	6.2/5.2	15
102	15/35	82-90	11	55	5.9/4.9	9
103	7/40	84-96	12	52	5.6/2.8	15
106	13/53	73-96	4.5	55	4.1/2.2	12
107	9/30	88-100	7.5	57	5.2/2.5	6

the plate tanks and were carried into the rinse tank with the drag-out. Essentially, the filter on the ED module was forced to carry the filtration load for the entire plating line. Filter runs were therefore undesirably short, averaging some 25 hours. Filter cartridges of several styles and pore sizes were tried without any marked improvement in the length of time between changes. A 25-micron cartridge was found to be the maximum porosity for acceptable filtration. Several bag filters were also tried with uniformly unsuccessful runs of only 1-2 hours operation before blinding. However, it was found that molded filter cartridges of resin-impregnated fiber could be cleaned by soaking in 10% hydrochloric acid and reused after rinsing in tap water. This minimized filter element replacement costs, but did not improve the clogging frequency. Similar cleaning of wound cartridges was not as successful. Eventually a higher capacity multiple-cartridge filter was installed, and filter runs increased to 60 hours. Later, an in-line strainer was added to remove any stray particles introduced by the handling or gradual deterioration of the reused cartridges. Independent and continuous filtration of the plate solutions, as usually employed on nickel electroplating lines, would substantially improve the operation of the ED prefilters and should be specified as a prerequisite for ED treatment.

A difficulty associated with the frequent filter clogging was the concomitant drop in flowrate through the ED stack that could go undetected by the operator who may be involved in other duties. If the fluid velocity through the feed cells decreases below the minimum required for adequate mixing at the operating current density, one or more of the cells may polarize. This phenomenon is caused by the formation of an ion-depleted layer at the membrane face, forcing the current to be carried by ionized water molecules that produce local variations in pH. In the case of nickel electrodialysis, the major polarization occurs at the cation-membrane, the feed solution becomes basic, and green nickel hydroxide precipitates in the flow path. Stack resistance increases, and the cell eventually becomes blocked. If uncorrected, adjacent cells become affected and performance deteriorates. The onset of polarization is indicated by increased voltage requirements across the stack (high stack resistance) and decreasing pH of the product solution. Increased pressure at the stack inlet may also occur. Examples are found, among others, in Table 2 data for days 45-46 and days 81-85. Remedial action in response to persistent signs of polarization consisted of recirculating a 5% acid solution through the stack for an hour or so and restoring the correct operating conditions. If polarization is allowed to proceed too far, the cells and membranes must be cleaned manually.

Blockage of the cell flow path or the inlet or outlet slots by particles contaminating the feed solution or misalignment or slippage of stack components can also induce polarization.

Slippage is almost always caused by overtightening the clamping bolts, especially when some shrinkage has occurred due to cold weather and/or unheated plating facilities and the stack is retightened to stop external leakage. Adjustments should always be made at room temperature, if possible.

Unit II data for the second series of tests are presented in Table 3. The Unit was operated on a second reclaim rinse in the same manner that Unit I functioned on the first rinse. After passing through Rinse I the plating barrel was given a second wash in Rinse II and then immersed in the destruct solution before spray rinsing the drying. Rinse II was not used to replenish Rinse I or the plate tanks, and except for occasional cleaning, water was added only to make up for evaporation losses. Rinse and product samples were again taken at the end of each day's operation. The average concentration in Rinse II was held to 0.38 g/l nickel with a product of 44 g/l nickel, a concentration ratio of 117. Since Unit II was required to remove less than one-tenth of the nickel that Unit I recovered it initially consisted of only 15 cell-pairs and operated at much lower current densities. However, it was found that variations in plating rate, drag-out volume, and Rinse I concentration caused considerable fluctuations in the amount of nickel carried into Rinse II and could require periodic operation at currents 2 to 3 times that shown in the Table in order to maintain a nickel concentration less than 500 mg/l. The value recorded in the Table is that at which the concentration remained reasonably constant at normal drag-out conditions as judged by conductivity readings. Under these conditions the volume of product collected from Unit II was approximately 600 ml/hour, and it was combined with the product from Unit I for return to the plate tanks.

The Rinse II pH was usually lower than that of Rinse I because of decreased removal of the H^+ ions which originated either from natural leakage of the very mobile H^+ ions across the anion-exchange membrane forming the anode compartment or from residual acid remaining after stack cleaning. Filter runs of many weeks were achieved with the Unit II prefilters even though 5-micron cartridges were employed. This is not surprising, since all drag-in solution to Rinse II had previously been treated by ED Unit I.

The major (only) problem encountered with Unit II during this period was the persistent formation of ferric hydroxide sludge in the product cells of the electrodialysis stack. This markedly increased stack resistance and necessitated operation at maximum available rectifier voltage (50v) in order to maintain a current sufficient to remove the nickel drag-in. Occasionally it was necessary to by-pass the second rinse for a few hours to prevent excessive nickel buildup. When the accumulation of sludge began to interfere with the normal flow of the product solution the membrane stack was flushed or left filled

TABLE 3. ELECTRODIALYSIS OF NICKEL RECLAIM RINSE II SECOND TEST SERIES.

Operating Day	Amps/Volts	Temp., °F	Feed, g/l Ni	Product, g/l Ni	pH Feed/Prod.	Time run, Hours
1	2/20	51-65	.27	35	7.2/6.1	2
2	1/13	60	-	-	-	4
3	2/18	60-70	-	-	-	7
4	4/36	65-72	.25	62	7.4/5.7	7
5	2.75/28	69-78	.18	62	7.4/5.8	9
6	2.5/08	61-75	.15	59	7.2/5.6	9
7	2.5/25	64-77	.22	60	7.2/5.8	9
8	2/37	38-64	.19	51	7.3/5.9	6
9	2/40	61-74	.16	60	7.0/6.1	9
10	2/48	63-71	.15	52	5.5/4.4	9
11	2/41	71-74	-	-	-	7
12	2/40	60	.17	47	3.7/1.4	5
14	2/28	66-69	.25	19	6.6/6.4	1
15	2/33	77	.25	56	6.4/8.0	8
16	2/36	68-78	.15	48	4.9/2.8	6
17	2/29	70-78	.09	42	3.9/2.4	5
18	2/23	73-74	.11	41	6.1/3.2	8
19	2/33	64-78	.13	53	6.5/5.2	8
20	2/20	72-79	.18	49	3.6/1.2	3
21	1.5/50	70-80	.24	48	5.6/3.6	8
22	2/45	60-72	.25	53	4.0/1.4	8
23	1/50	65-74	.28	50	4.9/2.8	7
24	1.8/50	62-83	.28	51	4.4/3.6	9
25	3.5/38	76-82	.15	54	3.1/1.6	6
28	2/20	65-72	.29	70	6.2/5.0	12
29	1/30	76	.16	51	7.4/6.0	8
30	1/30	70-82	.13	54	7.2/6.3	6
31	2/46	76	.33	66	7.2/5.4	8
32	2/47	80	.25	57	7.3/6.1	8
33	2/50	66-78	.34	61	7.2/6.0	9
34	1.5/50	60-76	.36	55	7.1/6.2	15
35	1.5/50	78	-	-	-	9
36	2/50	57-74	.66	20	6.6/6.3	15
37	1/50	72-76	.47	57	6.3/6.0	6

(continued)

TABLE 3. (continued)

Operating Day	Amps/Volts	Temp., °F	Feed, g/l Ni	Product, g/l Ni	pH Feed/Prod.	Time run, Hours
38	1.9/50	65-61	.73	66	7.2/5.7	12
39	1/50	61-70	.23	51	7.0/6.2	15
40	1.75/50	72-79	.21	52	6.8/3.9	15
41	1.75/50	76-79	.23	47	6.9/6.0	6
42	1.5/50	62-70	.24	50	7.2/6.0	10
43	1/50	58-68	.28	41	6.2/6.1	8
44	5/50	64-74	.46	35	6.7/6.2	9
45	1/50	68-76	.43	51	5.3/5.3	8
46	1/50	70-76	.48	42	6.2/5.2	9
47	.5/50	63-70	.31	33	5.8/6.4	9
48	.5/50	66-71	.27	28	5.6/6.6	10
52	1.5/50	66-73	.36	52	5.8/6.1	9
53	3/48	68-74	.20	41	4.2/2.1	14
54	1.5/28	67-72	.21	9	3.8/4.9	14
60	5/27	70-74	.35	53	7.6/2.8	12
61	4.5/24	64-79	.31	52	6.8/5.1	8
62	3/32	72-84	.16	31	5.4/2.3	9
63	2.5/38	74-82	.45	49	6.5/2.6	9
64	2/50+	78-80	.22	51	3.2/1.6	9
66	2/30	65-74	.38	32	3.9/1.9	7
67	2/40	66-75	.46	29	5.1/1.8	8
68	2/29	67-72	.37	33	5.4/2.3	17
69	2.5/36	70-78	.22	40	4.0/2.1	12
70	2/24	72-81	.54	22	4.1/2.4	9
71	1/23	63-69	.34	21	4.2/2.4	4
72	1.6/50	70-79	1.0	54	5.0/4.1	17
73	1.5/50+	73-82	.95	52	5.9/5.5	17
74	1/38	64-80	.47	48	6.2/6.1	14
75	1.75/26	68-74	.64	51	6.7/5.8	7
76	2/14	72-80	.49	61	3.3/2.5	17
77	1/50+	65-77	.47	37	5.4/4.5	8
78	1.5/50	74-78	.37	42	4.3/3.2	5
79	1.5/50+	68-80	.75	46	6.0/4.4	18
80	1/50+	77-82	.60	41	6.0/5.2	9

(continued)

TABLE 3. (continued)

Operating Day	Amps/Volts	Temp., °F	Feed, g/l Ni	Product, g/l Ni	pH Feed/Prod.	Time run, Hours
81	1.4/50+	72-76	.80	41	6.4/5.4	9
82	1.8/50+	72-79	.39	47	5.8/5.7	17
83	1/50+	73-78	.05	41	3.7/5.6	17
84	2/40	64-72	.15	38	3.3/1.8	6
85	2.8/46	69-72	.17	46	3.5/4.3	8
86	2/28	70-77	.22	48	3.8/4.6	15
87	1.5/33	71-84	.04	12	3.5/2.4	14
88	3/50+	73-88	.03	12	3.3/2.1	10
89	3.3/50+	70-82	.13	25	3.3/2.1	9
90	2/50+	73-84	.09	12	3.3/2.6	9
93	2/15	66-75	.25	16	3.1/2.5	9
94	2/45	77-89	.30	50	3.8/4.1	15
95	2/30	76-90	.29	38	4.0/4.6	8
96	1.7/32	80-92	.24	35	4.2/4.5	7
97	2/36	82-93	.29	35	4.1/5.1	7
98	1.5/32	70-85	.50	40	5.9/5.7	15
99	2.5/48	78-85	.90	50	5.9/5.7	15
100	3/50+	85-98	1.0	50	5.7/4.9	15
101	2.5/50+	86-89	1.2	51	5.8/5.1	12
102	5/30	82-88	2.0	49	5.5/5.4	10
103	2/35	83-92	1.2	34	4.5/5.7	15
104	2/42	92-95	.52	33	4.1/5.6	8
105	2/28	86-94	.40	28	5.1/5.4	15
106	2.5/46	72-85	.32	52	4.6/2.6	16
107	2/44	84-92	.58	38	5.2/3.9	6

overnight with 5-10% sulfuric acid, which usually restored the original stack conditions. The contamination of the product cells of Unit II was attributed to the fact that iron concentration in Rinse II was 15 times that in Rinse I, where no problem with sludge formation was encountered. This concentration difference was probably caused by the use of unlined stainless steel tank for Rinse II, while a PVC liner was installed in the Rinse I tank. Ferric ions in Rinse II thus had both a higher absolute and relative concentration and at the current densities used were concentrated in the product to the point of precipitation. The stainless Rinse II tank was coated with epoxy in an effort to limit iron contamination. An attempt was made also to maintain more consistent lower average current densities in order to limit the concentration of the product. Consequently, a 20 cell-pair stack was installed at the same time so that equivalent nickel removal could be maintained at lower and less variable amperages. These modifications appeared to be successful and permitted steady operation of Unit II during the final period of the field test, described in the next subsection. In addition to the formation of a precipitate in the product cells, it was observed on several occasions that the usual pH relationship of feed greater than the product solution was reversed to give higher values in the product. This was undoubtedly related to concentration changes caused by the precipitation in the product cells. In spite of the difficulties with sludge formation during this period, Unit II was operational 75% of the time plating was in progress, with an 85% availability for the last two months. Total operating time was 907 hours.

Third Series of Tests

Tables 4 and 5 give the data from the operations of Units I and II over the final six-month period of the field tests. Rinse I was held at an average concentration of 2.4 g/l nickel, and Rinse II was maintained at 0.37 g/l nickel. Results of the daily analyses of the recirculating rinse solution for each of the reclaim tanks are also plotted in Figure 7. The high concentration on days 4 and 5 in Rinse II and on days 11-14 in Rinse I were caused by increases in the plating rate without matching increases in the stack currents. Low Rinse II concentrations for days 27-30 were the result of omission of this tank in the rinsing sequence. The concentrations in both solutions are largely controlled by Unit I since increases in Rinse I concentration will produce a larger nickel drag-out to Rinse II. The ability of Unit II to compensate by increasing stack current density is limited by the necessity to avoid excessive concentration of iron in the product cells, as discussed earlier. In accordance with this constraint, the maximum current used on Unit II was 2.5 amperes. Coordinating the operation of Unit I and Unit II is therefore critical for maximum nickel recovery and/or minimum pollution from nickel-containing effluents. Average product

TABLE 4. ELECTRODIALYSIS OF NICKEL RECLAIM RINSE I
THIRD TEST SERIES

Operating Day	Amps/Volts	Temp., °F	Feed g/l Ni	Product, g/l Ni	pH Feed/Prod.	Time run, Hours
1	7/60	79-90	1.6	54	2.7/3.0	14
2	7/60	86-90	1.8	53	4.8/2.9	7
3	8/75	83-90	1.9	49	4.8/2.4	8
4	7/50	75-90	1.8	45	5.5/4.8	13
5	7/50	78-83	1.5	51	5.5/5.1	16
6	10/50	78-102	1.9	58	5.4/4.9	11
7	8/48	81-90	1.6	54	4.9/3.5	11
8	10/50	82-92	2.0	65	4.0/3.1	11
9	9/66	90-95	1.3	45	4.9/2.3	9
10	9/65	70-97	2.6	57	5.0/2.4	16
11	10/80	78-98	7.0	61	4.2/1.8	15
13	3/25	95	11.0	45	4.1/2.1	15
14	7/34	87	12.0	46	4.9/3.1	3
15	8/42	78-92	4.5	46	5.4/2.4	15
16	8/60	84-100	1.6	53	4.8/2.9	15
17	10/69	82-110	1.4	50	4.6/2.7	11
18	10/75	80-110	.89	62	3.5/2.6	16
20	9/90	80-110	1.3	59	4.6/3.6	7
21	12/52	90-97	3.0	69	5.0/3.9	6
22	9/60	74-95	1.6	68	5.6/4.6	10
23	8/60	78-96	1.3	69	5.5/4.7	11
24	10/50	81-95	2.6	66	5.4/4.6	7
25	5/85	77-92	5.0	45	4.9/1.3	16
26	11/47	82-89	3.5	68	4.1/2.9	12
27	10/45	80-94	2.3	63	5.0/3.9	10
28	9/50	80-88	1.9	59	5.2/4.2	8
29	9/49	80-82	2.8	61	5.4/4.6	8
30	7/42	69-82	3.0	52	5.5/4.7	9
31	10/50	68-86	2.9	66	3.7/3.0	9
32	8/50	75-86	3.0	66	5.3/2.1	9
33	9/62	73-83	4.0	50	5.1/2.7	9
34	9/51	65-81	2.2	53	4.4/2.1	7
35	9/50	68-87	2.1	42	6.1/4.2	9
36	8/40	73-87	2.1	43	6.1/4.6	9
37	7/39	78-86	1.9	40	6.2/2.8	5

(continued)

TABLE 4. (continued)

Operating Day	Amps/Volts	Temp., °F	Feed g/l Ni	Product, g/l Ni	pH Feed/Prod.	Time run, Hours
38	8/45	74-80	2.6	37	6.5/1.9	4
39	8/50	63-86	2.4	43	4.2/2.5	8
40	6/36	70-90	2.5	44	6.0/1.9	15
41	8/40	74-92	1.6	54	3.7/1.7	15
42	9/40	84-88	1.5	57	5.6/1.5	10
43	7/50	78-90	1.9	59	5.7/1.5	15
44	7/60	66-88	3.2	59	6.2/1.2	15
45	7/34	81-88	3.3	56	6.0/2.1	9
46	8/43	75-82	2.6	56	4.0/2.8	7
47	8/50	64-79	1.7	49	4.6/4.3	15
48	8/47	80-85	2.6	49	5.0/4.6	9
49	8/43	62-78	3.2	43	5.0/5.4	5
50	8/50	70-82	2.4	58	5.5/1.6	7
51	8/45	76-84	-	-	-	9
52	5/59	70-90	2.0	56	4.2/1.8	12
53	8/46	70-80	2.4	45	4.2/2.8	9
54	8/55	72-79	1.7	58	4.8/3.0	9
55	8/60	79-88	1.5	59	4.8/3.1	8
56	6/38	70-82	2.0	50	5.4/3.3	9
57	8/52	74-86	2.2	62	4.6/3.1	9
58	8/55	78-88	2.4	57	4.6/3.4	9
59	7/47	60-84	2.3	56	5.2/1.9	9
60	7/65	69-84	2.4	61	4.8/1.9	9
61	7/55	62-77	2.5	46	5.7/1.7	4
62	9/70	74-80	3.0	61	5.5/1.9	9
63	7/74	64-84	1.8	57	5.2/4.2	7
64	3/45	67-76	2.0	-	6.3/-	3
65	7/73	82-90	2.3	50	5.2/3.5	9
66	6/33	71-90	1.9	44	4.6/2.9	7
67	6/57	75-78	1.9	48	6.1/4.7	9
68	10/76	60-80	-	46	-/5.2	9
69	5/30	62-72	1.2	33	2.9/2.3	5
70	6/44	75-85	1.5	37	5.0/3.2	10
71	9/36	83-92	2.5	52	6.1/5.2	9
72	8/35	74-90	1.7	43	4.7/2.6	8

(continued)

TABLE 4. (continued)

Operating Day	Amps/Volts	Temp., °F	Feed g/l Ni	Product g/l Ni	pH Feed/Prod.	Time run, Hours
73	7/33	70-92	1.7	39	5.9/3.2	9
74	7/30	72-86	2.4	41	5.9/4.8	15
75	8/35	-90	2.2	45	6.0/4.9	9
76	6/29	70-77	2.3	37	6.3/5.4	3
77	9/35	64-89	2.5	46	3.2/2.3	7
78	8/40	72-89	2.0	44	4.7/3.1	9
79	8/54	76-89	1.9	47	5.2/3.8	8
80	10/53	76-92	2.3	53	5.4/4.3	9
81	7/50	76-90	2.0	41	4.0/2.6	9
82	5/43	76	1.7	47	6.0/3.6	3
83	6/60	67-86	2.0	49	6.4/5.6	8
84	8/56	74	2.4	30	6.9/6.0	3
85	10/73	68-88	2.0	61	6.8/5.8	9
86	10/63	78	2.3	45	7.0/5.7	3
87	6/35	64-70	2.5	47	7.0/5.8	4
88	7/40	74-84	2.7	50	7.0/5.7	9
89	5/50	57-79	2.1	43	6.2/3.9	6
90	9/97	82	2.3	61	5.7/3.5	9
92	8/43	63-78	2.8	50	6.9/5.8	7
93	8/65	68-82	2.0	49	6.9/6.1	9
94	9/65	80	2.6	48	7.2/6.1	4
95	8/80	84-89	2.7	42	7.1/6.8	9
96	7/50	74-84	2.6	69	6.7/5.4	6
97	12/87	72-92	3.4	78	6.7/5.7	9

TABLE 5. ELECTRODIALYSIS OF NICKEL RECLAIM RINSE II
THIRD TEST SERIES

Operating Day	Amps/Volts	Temp., °F	Feed g/l Ni	Product, g/l Ni	pH Feed/Prod.	Time run, Hours
1	1.8/10	81-88	.31	18	4.2/3.5	7
2	1.6/10	83-86	.38	21	4.3/3.5	7
3	1.5/10	81-85	.35	20	4.7/3.8	8
4	2/9	72-86	1.30	31	5.5/4.1	13
5	2/9	78-90	1.46	32	5.3/4.8	16
6	2/23	80-93	.17	21	3.6/3.7	16
7	.5/2	81-85	.46	11	4.6/3.6	11
8	1/4	80-89	.39	20	4.9/3.7	11
9	1/8	70-83	.26	28	5.2/4.0	16
10	1.5/10	72-88	.34	29	4.7/3.6	16
11	1.5/10	74-94	.70	29	5.1/3.8	15
12	1.5/21	78-93	.48	30	4.0/3.8	15
13	2/36	80-95	.38	35	4.5/3.6	16
14	2.5/38	74-81	.75	38	4.7/3.7	6
15	2/35	78-90	.71	43	4.4/3.5	15
16	2/40	82-94	.29	33	3.7/3.5	15
17	1.8/25	82-96	.26	33	4.1/3.4	11
18	1.8/22	88-96	.28	31	4.2/3.3	13
19	1/26	80-88	.31	21	4.6/3.6	5
20	1.8/28	76-90	.29	34	4.0/3.5	10
21	1.4/25	80-88	.37	30	3.7/3.6	9
22	1.5/12	76-90	.29	19	4.5/3.6	10
23	1/15	76-92	.21	23	4.7/3.8	11
24	1/14	81-88	.41	45	4.9/4.0	7
25	1/19	79-90	.25	29	4.9/3.9	16
26	1/12	79-89	.45	29	4.9/3.9	8
27	2/22	78-90	.10	24	2.8/2.0	9
28	1/5	82-86	.05	11	2.9/2.4	8
29	.8/6	76	.03	8	3.2/2.5	7
30	.5/4	71	.05	-	3.2/-	7
31	1/16	71-84	.44	25	4.1/2.7	9
32	1/15	78-80	.52	25	4.4/3.2	9
33	1/24	70-77	.57	26	4.4/3.5	6
34	1.5/9	64-77	.53	22	2.6/2.3	10

(continued)

TABLE 5. (continued)

Operating Day	Amps/Volts	Temp., °F	Feed g/l Ni	Product, g/l Ni	pH Feed/Prod.	Time run, Hours
35	1/11	72-86	.12	21	2.9/2.3	13
36	.8/6	72-84	.19	16	3.7/2.9	9
37	.8/7	66-84	.43	12	4.5/3.6	9
38	.5/7	74-76	.46	13	4.8/3.3	6
39	.5/18	66-82	.50	15	5.0/3.8	9
40	1/36	73-86	.53	31	2.3/3.1	15
41	1/36	78-85	.29	28	4.1/3.0	15
42	.5/17	81-84	.38	20	3.0/3.2	10
43	.5/40	75-78	.31	25	2.6/3.0	15
44	.5/37	68-86	.31	22	2.6/2.8	15
45	.5/20	76-85	.39	17	3.1/3.3	9
46	2/10	72-78	.28	19	2.8/2.1	6
47	1/12	74-75	.28	17	3.2/2.5	15
48	.8/20	74-78	.21	13	3.5/3.0	8
49	.8/29	72-72	.22	8	3.4/3.5	5
50	.8/28	70-80	.23	15	3.4/4.0	9
51	.5/25	72-80	-	-	-	9
52	.5/17	66-76	.51	19	4.8/4.6	13
53	.5/18	72-76	.55	12	5.0/4.4	7
54	.5/20	71-74	.54	21	5.1/4.5	15
55	1.5/10	68-72	.03	8	3.2/2.1	4
56	.3/3	70-83	.16	16	3.3/2.6	9
57	.5/3	74-83	.32	11	2.8/2.2	9
58	.5/2	76-74	.36	13	3.3/2.5	9
59	.5/3	60-70	.39	12	3.1/2.1	9
60	1/6	60-75	.42	25	3.2/1.9	9
61	.5/4	66-75	.32	18	3.3/2.1	9
62	.8/7	73-74	.33	23	3.4/2.2	9
63	.8/7	66-74	.32	21	3.4/2.3	8
64	.8/6	65-68	.39	-	3.6/-	4
65	.8/7	78	.33	17	3.7/2.5	9
66	.8/6	77	.36	21	4.1/2.7	6
67	.6/6	72-74	.37	17	4.5/2.8	9
68	.7/8	64-72	.35	20	4.8/3.0	9
69	.8/7	65-72	.36	21	3.8/2.6	5
70	.6/8	71-74	.32	24	3.9/2.7	10

(continued)

TABLE 5. (continued)

Operating Day	Amps/Volts	Temp., °F	Feed g/l Ni	Product, g/l Ni	pH Feed/Prod.	Time run, Hours
71	.7/8	72-76	.37	20	3.9/2.8	9
72	.9/7	68-76	.35	17	3.8/2.9	8
73	.5/5	76-77	.32	16	3.8/3.1	9
74	.5/11	70-78	.30	26	3.4/2.7	12
75	.5/7	75	.32	24	3.5/2.9	9
76	1.5/14	65-68	.37	12	3.3/2.3	3
77	1.9/25	74-75	.48	30	3.3/2.3	9
78	2.5/10	66-70	.31	29	2.5/1.6	13
79	1.5/7	68-72	.12	18	2.6/1.9	9
80	.5/21	65-72	.17	13	3.0/2.0	5
81	1/15	68-75	.25	18	2.9/2.1	9
82	.75/13	72	.22	18	2.9/2.1	4
83	1.8/6	67-74	.29	32	3.0/2.2	8
84	1.4/7	66-70	.29	37	3.2/2.3	9
85	1/7	68-70	.29	36	3.3/2.3	9
86	1/7	72-76	.29	45	3.4/2.5	9
87	1.4/8	68-72	.41	23	3.9/2.6	6
88	.8/9	70-74	.42	35	4.1/2.7	9
89	1/7	57-68	.38	12	3.8/1.9	8
90	1.5/8	70	.44	17	3.4/2.1	9
91	1.4/11	74	.40	25	3.6/2.4	9
92	1.1/19	67-74	.44	28	3.7/2.4	8
93	.9/13	68-74	.49	33	3.7/2.9	9
94	.9/15	74	.51	19	4.1/2.6	9
95	.5/16	76	.64	30	5.2/4.9	9
96	1/6	72	.49	10	3.5/2.7	7
97	.6/5	68-78	.54	23	4.3/2.7	9

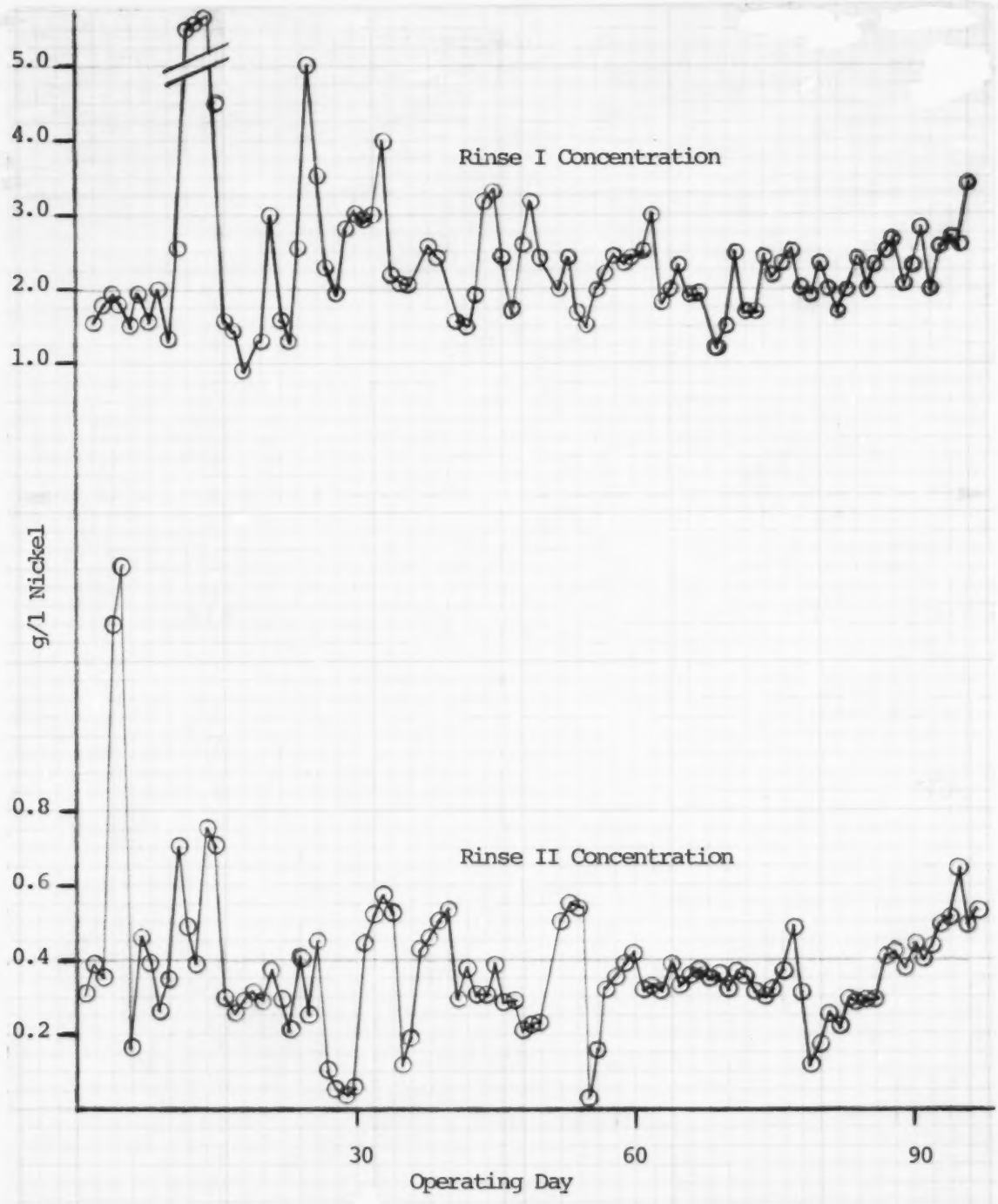

Figure 7. Rinse concentrations for third test series.

concentrations were 52 g/l nickel and 23 g/l nickel metal in Rinse I and Rinse II, respectively. Corresponding concentration ratios were 22 and 61. Volume of concentrated nickel product solution from each of the ED Units was the same as in the previous operating period for a total of about 45 liters (12 gallons) returned to the plate tanks per eight-hour day. Product concentrations were lower than for the preceding period because improved operation of the plating line with concurrent continuous nickel recovery permitted electrodialysis treatment at lower and more uniform current densities. Nickel drag-in was less and and had less fluctuation than in Periods I and II. Unit I operated 96% of the time the plating line was running, and Unit II was on-line 98% of the time. These percentages would have been 98% and 100% if operators were available at all times. Operating time was 858 and 933 hours, respectively for Units I and II for totals of 2,225 and 1,840 hours over the entire demonstration program.

The excellent availability of Unit II was attributed to the epoxy lining applied to the Rinse II tank at the end of the last operating period and the ability to operate at the lower current densities. In addition, intermittent flushes with dilute sulfuric acid were used as a routine maintenance procedure, and no further problems were encountered with sludge formation in the product cells. Only one episode of pH reversal was observed during this period. Inspections of the stack showed it to be clean and clear with all passages unblocked and free of solids or deposits on the membranes. At an inspection near the end of the period the two electrode membranes showed substantial erosion on the side facing the electrodes after 1,613 hours operation. This was normal attrition resulting from attack by the oxygen and/or chlorine formed at the anode and partially recirculated with the electrode solution. Use of a degasser in the electrode reservior and heavier (i.e. thicker) membranes would improve the life of these electrode membranes. Replacement rates for the other stack membranes were 39% for Unit I and 15% for Unit II over the total time of the field test. Considering the experimental nature of the demonstration program, this seems to indicate that membrane usage in a fully developed system will be comparable to the 20% rate commonly used for estimations of membrane requirements in commercial electrodialysis installations.

Unit I was also available a high percentage of the plating time during this period. Installation of larger prefilters and an in-line strainer immediately before the stack, mentioned earlier, minimized any cell blockage caused by sludge or particles from the rinse tank or pretreatment operations and extended the filter runs. Intermittent decreased flow rates through the stack, although less frequent, were the chief difficulty encountered. As before, these episodes were caused by the slow buildup of material on the filter cartridges or by failure to

return to normal flow rate after a cleaning or warmup period. An acid flush was generally successful in restoring satisfactory operating conditions. In light of this experience, future units should incorporate a pressure transducer set to open the stack DC circuit if pressure/flow drops below a predetermined minimum. Design improvements are also possible which will minimize flow variations and opportunities for maldistribution of feed solution. A small expansion tank was installed on the product line during this period. This retained a sufficient volume of product solution to keep the product cells full as they expanded when the pressure on the feed cells was removed at shutdown. Otherwise air could be sucked into the product cells and contribute to some of the observed voltage increases.

Drag-out volume on the demonstration nickel line was calculated at an average of 2 liters per barrel from the rinse solution volumes and bath analyses before and after rinsing. The usual plating rate was about 2 barrels per hour for 12 hours (1.5 shifts) a day. If an 80% nickel sulfate and 20% nickel chloride bath composition is assumed, at the operating conditions and rinse tank concentrations maintained during the final period of the demonstration program only 19 kg (41 lbs.) of nickel salts per year would be lost as drag-out to the destruct system, in contrast to an estimated 3,540 kg (7,800 lbs.) of salts without electrodialytic recovery. The normal salt usage figure is based on the average of estimates derived from reclaim solution concentration without ED, the plater's salt addition schedule, and Risdon's recent gross purchasing record. Although Risdon's inventory procedures do not permit identification of individual plating line requirements, observations by the operator tended to support such usage reductions. They indicated that several 2-3 week periods with no plating salt additions were experienced on the test line during the early part of the field test, and that during the final test period nickel salt additions were necessary only after the first 3-1/2 months and then only after solution losses due to tank cleaning. Plating salt addition requirements were based on quality control inspection of plated work and independent analyses by Risdon's laboratory. Of the estimated 3,521 kg of nickel salts saved, 97% were recovered by Unit I. Unit II, therefore, functioned primarily as a pollution control device by further reducing the amount of nickel eventually going to destruct and not as a nickel recovery apparatus. It was designed to achieve, as closely as possible, a closed-loop plating operation. To this end, it appears entirely possible to further reduce the nickel concentration in Rinse I to around 1.0 g/l nickel and in the final rinse to less than 100 mg/l by appropriate design and sizing of the ED modules. This would cut the nickel salt loss from the demonstration line to less than 4.5 kg (10 lbs.) per year.

Besides the savings in plating salts, the low nickel loss resulting from the combined recovery would produce a reduction of 1,317 kg (2,900 lbs.) per year, on a dry basis, of nickel hydroxide precipitated from the destruct reaction. This would have to be disposed of as 13.3 m^3 (3,500 gallons) of a 10% solids sludge. If sodium hydroxide alone is assumed as the precipitant, destruct chemical requirements are reduced by 1,180 kg (2,600 lbs.). In all, over 98% of raw materials and destruct chemicals are saved via the ED treatment.

A unique additional advantage was realized on the demonstration plating line because of the use of the inorganic brighteners cobalt sulfate and cadmium chloride. Since they are ionic, they were also concentrated by the electrodialysis equipment about ten-fold from the rinse to the product solution and returned to the plate tanks along with the recovered nickel. As a result, the addition of brighteners to the plate solutions could be cut by about 25% for an additional raw material savings. Similarly, almost any ionic constituent in the plating solution will be concentrated, at least to some extent, by electrodialysis treatment.

SECTION 6

COST ESTIMATE

An estimate of the savings possible through recovery of nickel from plating rinses by electrodialysis is presented in Table 6. The costs are based on operation of a Watts-type bath similar to that of the demonstration plating line at Risdon but using an increased work rate of 4 barrels per hour and two shifts per day. Only a single electrodialysis unit operating on Rinse I, the reclaim tank immediately following the plate tanks, is considered since it accounts for 97% of all metal values recovered. An average concentration of 50 g/l nickel in the reclaim tank is assumed without use of electrodialysis recovery. This is consistent both with computations based on standard rinsing formulae and with actual analyses of the Risdon reclaim tanks.

Calculation of the amount of nickel salts lost was done by attributing the total nickel content of the plate solutions to nickel sulfate or nickel chloride on an 80:20 weight ratio. Costs of $0.74 per pound for the sulfate salt and $1.04 per pound for the chloride were used. The amount and cost of destruct chemical were calculated on use of only sodium hydroxide at $0.29/lb. as precipitant for the nickel. Figures for sludge disposal are for a 10% solids solution removed by a contract hauler to a treatment/disposal site within 50 miles. Various factors for relating quantity of nickel lost to treatment chemicals and sludge volume were taken from Graham (7). Savings realized by nickel recovery were determined by the difference between the amount of metal lost with and without recovery. Total savings of nickel salts, destruct chemicals, sludge disposal, and brighteners are estimated at $16,421/year or $68 per operating day. Recovered nickel salts and the corresponding reduction in destruct chemicals provide an annual savings of $13,451. On plating lines using countercurrent rinsing only, with no reclaim tanks, potential savings are substantially greater. Additional benefits may be realized from less rinse water usage, decreased handling and storage of raw materials, maintenance of optimum rinse concentrations, and decreased maintenance on and size of the required destruct system. If operating time is increased to three shifts or rate of work movement to the more usual 5 or 6 barrels per hour, potential savings will also increase. Production increases of this magnitude can be accomplished with little or no

TABLE 6. ESTIMATED COST SAVINGS USING ELECTRODIALYSIS FOR NICKEL RECOVERY

Assume: Reclaim tank= 50 g/l average nickel concentration
Plating rate= 4 barrels/hour; 2 liters/barrel dragout
Reclaim tank with ED= 2 g/l nickel
Two shifts; 240 days/year

	Without recovery	With recovery
Nickel as metal lost, pounds/yr	3,383	135
Nickel salts lost, pounds/yr	14,875	593
Value of nickel salts lost, $/yr	11,830	472
Caustic for destruct, pounds/yr	5,061	202
Cost of caustic, $/yr	1,468	59
Sludge for disposal, pounds/yr	5,345	213
Cost of sludge disposal, $/yr	712	28
Total cost of nickel lost, $/yr	14,010	559
Sludge decrease, pounds/yr	5,132	
Savings on recovered nickel, $/yr	13,451	
Savings on brighteners, $/yr	2,970	
Total savings per year	$16,421	

additions to the ED equipment required for the example given.

If pollution control is of primary importance and it is desired to minimize the metal discharged from the plating line, a second ED unit can be used to treat a second reclaim tank as was done in the demonstration program. If this second rinse is added to the recovery system and is held at 0.3 g/l nickel by Unit II, only 41 kg (90 lbs) per year of nickel salts will be lost, generating an additional savings of $400. If a 50 mg/l concentration is maintained in the second rinse, nickel salts lost to the waste disposal system would be reduced to 7 kg (15 lbs).

Estimated operating costs for Unit I only are given in Table 7. Electric power includes pumping and stack DC at 2.5¢/kWhr. Labor is based on three-quarters of an hour a day for plating room personnel to start-up and shut-down the units plus intermittent adjustments. Most of the time is required at start-up when the unit must be brought on-stream gradually, but at no time is continuous attention necessary. Filter costs are for replacement cartridges and probably conservative in view of the demonstrated possibility of cleaning and reusing them. Membrane costs are based on 20% replacement per year, the usual rate used in desalting equipment. Simultaneous operations of Unit II, if used, would add approximately 25% to these costs. Minimum net savings by use of one electrodialysis unit after subtracting the operating costs total $14,829 per year or $11,859 based only on nickel salts recovered. Capital costs will vary with drag-out rate, specified concentration in the rinse tank, and instrumentation supplied with the unit, but in most cases estimated payback for purchase of Unit I only would be 18 months or less even if based on the lower dollar savings of recovered nickel salts alone.

TABLE 7. ESTIMATED OPERATING COSTS FOR 50 CELL-PAIR NICKEL RECOVERY UNIT

		$/day	$/year
Electric power		.70	168
Labor		3.75	900
Chemicals		.02	5
Filter cartridges		.91	219
Replacement membranes		1.25	300
	Total	$6.63	$1592

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